Synchrotron x-ray diffraction using triple-axis spectrometry. Progress report for 1980

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SYNCHROTRON X-RAY DIFFRACTION USING TRIPLE-AXIS SPECTROMETRY

Progress Report for 1980

Jens Als-Nielsen

Abstract. High resolution X-ray diffraction studies of (i) monolayers of the noble gases Kr and Ar physiosorbed on graphite (ii) smectic A fluctuations in the nematic and the smectic A phases of liquid crystals are described. The apparatus used is a triple axis spectrometer situated at the storage ring DORIS at Hasylab, DESY, Hamburg. A monochromatic, well collimated beam is extracted from the synchrotron radiation spectrum by Bragg reflection from perfect Si or Ge crystals. The direction of the beam scattered from the sample is determined by Bragg reflection from a perfect Si or Ge analyzer crystal. High intensities even with resolution extending beyond the wavelength of visible light can be obtained.

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1. INTRODUCTION

The broad wavelength band of intense electromagnetic radiation associated with the centripetal acceleration of electrons in a storage ring extends well into the X-ray region for typical storage rings like DORIS at the DESY laboratory in Hamburg. The superrelativistic electron energy E implies that the radiation is confined to the plane of the electron orbit within an angle of $m_o c^2/E$. A monochromatic beam obtained by Bragg reflection from a perfect Si (1,1,1) crystal face placed in the white beam after a 1 mm wide slit at a distance of 20 meters from the electron orbit will have approximately the same band width as the natural width of the $CuK_{\alpha 1}$ line from a conventional X-ray tube, but for identical monochromators at a storage ring (E = 4.3 GeV, I = 50 mA) and at an X-ray tube, the monochromatic beam from the former is more than a hundred times more intense than that from a 10 kWatt rotating anode tube, and at the same time the vertical resolution is typically 25 times better! These numbers have been derived by experimental comparison between our set-up at DORIS Hasylab and at the 12 kW rotating anode at Risø. Furthermore, the wavelength as well as the monochromatic band width can be varied at will at the synchrotron by changing Bragg angles, apertures and monochromator crystals. With the accessibility of storage ring X-ray sources, so-called synchrotron radiation, the limits of X-ray diffraction methods have suddenly been extended by several orders of magnitude and new perspectives in experimental condensed matter physics are appearing.

In realizing the potential possibilities of synchrotron radiation, Professor Buras and I started a couple of years ago negotiations with the synchrotron radiation group at DESY in Hamburg about building a very flexible instrument for X-ray diffraction, known as a triple-axis spectrometer, at one of the beam lines of the planned synchrotron radiation laboratory, HASYLAB. Professor Buras and his collaborators have mainly been interested in the development of the energy dispersion method and this activity is reported on in a separate paper.

The triple-axis spectrometer project was generously accepted by the German scientists, and the instrument was funded by the Danish Natural Sciences Research Council [1] and Riso National Laboratory. Some parts of the instrument were produced in Physics Laboratory II, University of Copenhagen. We also realized the necessity of carrying out initial studies at the home basis laboratory Risø before taking the experiment to the synchrotron source, a conjecture that turns out to be very practical indeed, and a rotating anode source [1] was funded as well as auxiliary X-ray diffraction equipment [2].

Before actual experiments can be carried out successfully the new technique requires experience in a whole set of unconventional problems. These include monitoring of the position and the intensity of the incident white beam, suppression of higher order contamination in the monochromatic beam, and in establishing an acceptably low background count rate. Furthermore, the relatively short periods of beam time available for synchrotron radiation in a foreign laboratory requires that the time spent on line-up of the precision instrument should be minimized and the actual accumulation of data should be as effective as possible.

It was therefore a great help in addressing these problems that we during the construction period of HASYLAB in 1979 had access to synchrotron radiation in the European Molecular Biology Laboratory (EMBL) outstation at DESY. Drawing on the experience gained here we were ready to set up a preliminary version of the permanent beam line D4 in HASYLAB, install the instrument and carry out several experiments during a 4-week period in October-November 1980. In this report we give a description of out present set-up in HASYLAB followed by a brief outline of three experiments completed in the above mentioned period. The work was carried out in collaboration with a number of scientists from Denmark and U.S.A. as well as members from the technical staff of Risø as indicated by the names and addresses below the titles of the following sections.

2. EXPERIMENTAL SET-UP

J. Als-Nielsen, J. Linderholm, S. Jorgensen, P. Skaarup, and E. Dahl Petersen, Riso National Laboratory and B. Buras, Physics Laboratory II, University of Copenhagen.

A schematic top view of the set-up is given in Fig. 1. The synchrotron beam is travelling in a 20 m long, high vacuum tube terminated by a Be window, which has He gas at atmospheric pressure on the other side in order to avoid corrosion from radicals formed in air by the intense beam. The beam is limited to 1 mrad in the horizontal direction by a water-cooled Cu aperture at approximately 14 m from the source point. The beam is cut down to 0.1 to 0.2 mrad by a Pb beam definer in front of the monochromator and a Pb shield around the monochromator serves as an effective stop and trap for the powerful white beam.

The monochromatic beam is trimmed by a set of horizontal and vertical slits and monitored by an ionization chamber filled with 0.1 atm. of argon and methan mixture giving a transmission of approx. 95% at 1.5 Å. The current from the ionization chamber is converted to voltage by a Keithly amplifier (10^3 to 10^{11} V/A) and converted to a count rate by a CAMAC voltage to frequency converter. The wavelength of the monochromatic beam can be changed by scanning the Bragg angle θ_M of the monochromator. A convenient wavelength calibration is furnished by scanning θ_M while monitoring the fluorescent yield from a piece of Fe in the monochromatic beam. The yield increases discontinuously as one passes through the K-edge of Fe at 1.74334 Å.

The sample table mounted on the monochromator arm is centered in the beam by scanning the arm and watching the TV picture of the shadow of a pin on the table on a fluorescent screen behind the pin. The sample table is equipped with two horizontal translation stages and two perpendicular goniometer arcs (-20° to $+20^\circ$).

The analyzer table is identical to that of the monochromator. Evacuated Al tubes with Pb lining on the inside and Be windows in the ends are inserted between the sample and analyzer and

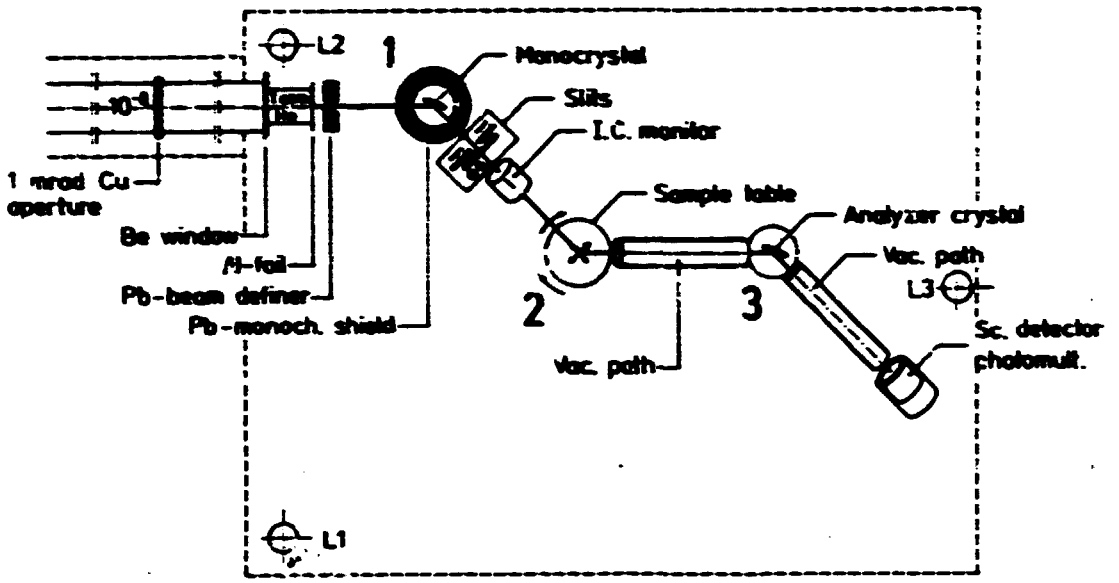

Fig. 1. Top view of the triple axis spectrometer. The 3 axes marked 1, 2 and 3 are the monochromator, the sample and the analyzer axes. The dashed line indicates the Al-Pb-Al walls of the hutch necessary for radiation protection.

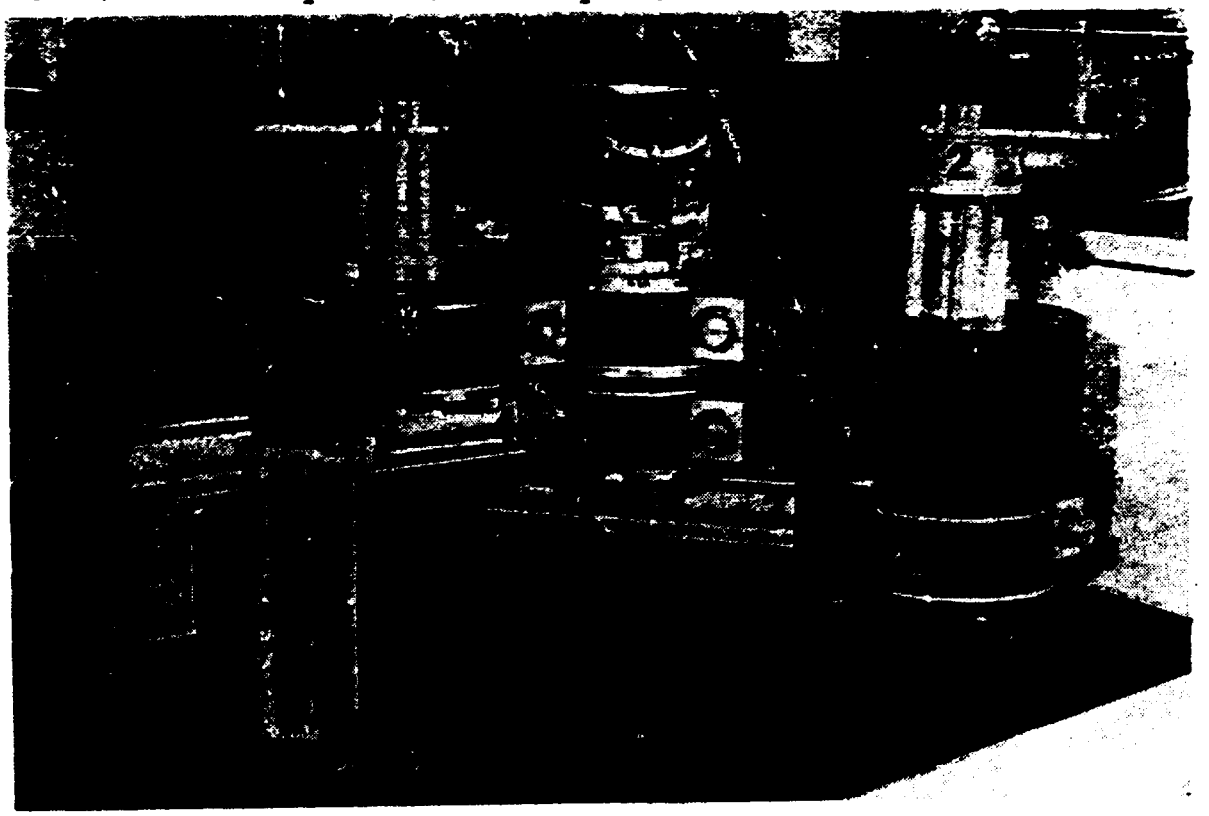

Fig. 2. Monochromator, sample and analyzer axes are marked 2, 5 and 6 respectively. Note the possibility of sliding the axes on the arms (3, 4 and 7) and the air cushion 8.

between the analyzer and the detector in order to minimize intensity loss by air absorption. The latter beam path has another very important feature: it limits the detector solid angle to a few millisteradians aimed right at the analyzer crystal, so the background count rate can be kept as low as 0.5 c/sec even at E = 4.3 GeV in the rather high general radiation level present around the spectrometer, when the beam is on. The entire spectrometer is situated in an enclosed hutch with Al-Pb-Al sandwich walls for radiation protection. The spectrometer can be viewed with beam on through a Pb-glass window.

A photo of the spectrometer is given in Fig. 2 [3]. The spectrometer arms are supported by air cushions supplied only by compressed air while the corresponding motor is running. The distances between the axes can easily be varied as the arms are made of convenient Aluminium profiles.

For certain experiments the analyzer is not necessary and the whole analyzer system can readily be detached from the spectrometer.

The entire floor supporting the spectrometer can be adjusted to beam height by turning the three spindle legs L1, L2 and L3.

All turn tables (Huber) are driven by Slo-Syn stepping motors with 1 step = 0.001° except the monochromator and analyzer tables where 1 step = 0.0005° so a 1:2 ratio between table and a arm is readily obtainable. The system is operated from a Decwriter terminal connected to a PDP11/34 computer with dual floppy discs. The computer is interfaced to a CAMAC system containing stepping motor modules, scalers, digital voltmeter interface and a limit switch module. Programs for executing measurements are written in Basic [4]. On the Decwriter the results and a coarse plot of the data appear on-line.

3. EXPERIMENTS

A. Two-Dimensional Structure of Kr and Ar Physiosorbed on Graphite

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The physics of condensed matter systems in lower dimension than three is currently an area of research of considerable interest and effort, theoretically as well as experimentally. In substrate systems graphite turns out to be a uniquely smooth substrate, and in certain graphite products substantial physiosorbing surface area per volume unit can be obtained. In the present case, using so-called UCAR-ZYX, the coherence length might be as large as several thousands Ångstrøm so the observed structure may not be seriously affected by more or less random boundary conditions as the coherence area will in that case contain of the order of a million atoms. In order to take full advantage of the large coherence area a high resolution scattering instrument is necessary, and as we shall see synchrotron radiation using Ge (111) as monochromator and analyzer at a wavelength of about 1.5 Å yields the sufficient intensity at narrow resolution for such studies.

Our first aim was the determination of the coherence length in our graphite substrate cell. To that end a monolayer of Kr atoms were physiosorbed in the cell. Although graphite is smooth there is of course a shallow potential well at the centre of each carbon hexagon in the graphite honeycomb lattice where a noble gas atom preferantially will be physiosorbed. For Kr the Kr-Kr interaction potential favours a near-neighbour distance quite close to that obtained when a Kr atom is physiosorbed in every third of the small honeycomb cells. For a complete monolayer the Kr lattice is thus in registry with the honeycomb lattice (the so-called $\sqrt{3}$ structure) and the sharpness of the Bragg peak from the Kr lattice gives the coherence length. However, the high-wavevector side of the Bragg peak is intrinsicly broadened in a

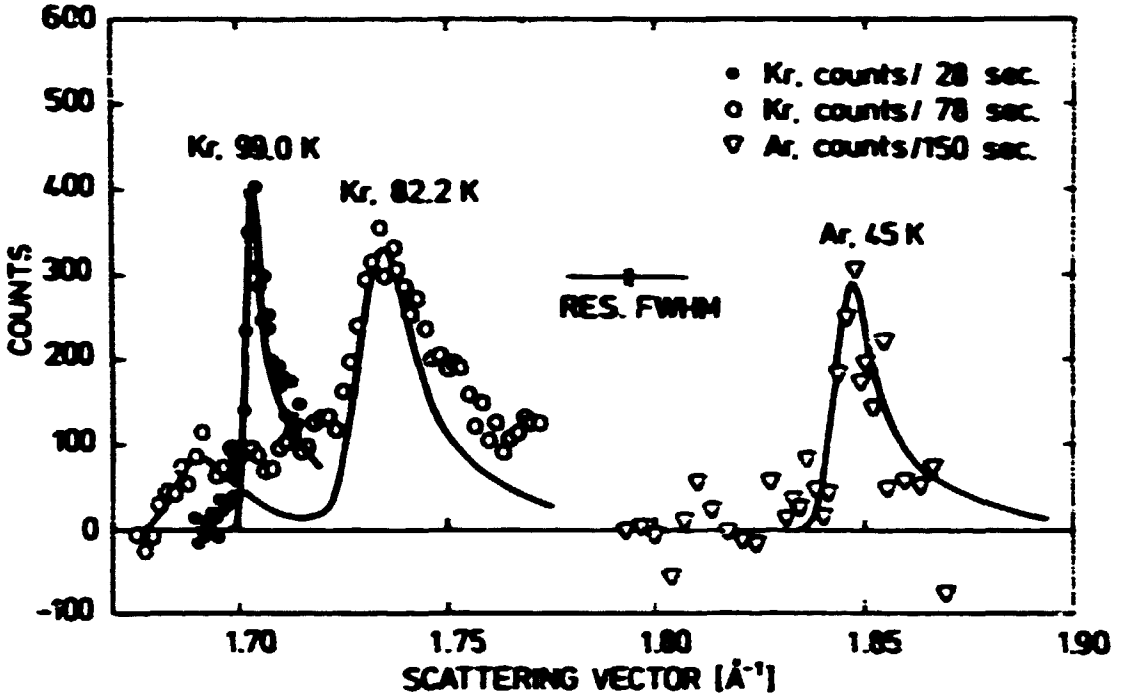

Fig. 3. Diffraction from the (1,0) peak of a 2 dimensional "powder" of a noble gas physisorbed on a graphite substrate, ZYX. For Krypton the structure may be in registry with the graphite honeycomb lattice (filled circles) and the sharpness of the low-wavevector part of the peak gives the coherence length (2000 Å) of the substrate. At lower temperatures there are more Kr atoms than honeycomb sites and the incommensurate diffraction peak is broadened significartly (open circles). For Ar the structure is incommensurate at all fillings and temperatures. Nevertheless the diffraction part is relatively sharp corresponding to a coherence length of 750 Å.

2-dimensional powder [6], so the coherence length is actually determined by the sharpness of only the low-wavevector side of the Bragg peak.

The left part of Fig. 3 shows the (1,0) diffraction peak of the $\sqrt{3}$-Kr structure (full circles). The full line is a least squares fit of the Bragg line profile with the coherence length L as an adjustable parameter. The best fit value is L = 2000 Å.

The open circles show the intensity profile for the same Kr filling, but now cooled down to 82 K. At this temperatur there are slightly more physisorbed Kr atoms than available sites in a registered structure. There are several possibilities for accommodating the extra Kr atoms. Imagine for example that the graphite honeycomb lattice is divided into 3 equivalent sublattices, say A, B and C. In registry only one sublattice is occupied. A slight overfill might give rise to a structure where the majority of atoms are still in registry now occupying say the A lattice in one domain, and the B and C lattices in neighbouring domains. The domain structure could be a super-honeycomb lattice, the extra atoms being accommodated in the domain walls. The full line is the structure factor for such an arrangement now with a coherence length of 500 Å. The data are not consistent in detail with the domain model and other models should be investigated.

Our results for Kr are idential to those obtained by Moncton, Birgeneau and cowcrkers [7] a few months ago at the Stanford storage ring, both as far as the quality of substrate, resolution and intensities are concerned. Our colleagues plan to study the incommensurate structure of Kr in more detail at SSRL, so we decided to utilize the technique to study a fundamentally different system, that is Ar which is incommensurate with the honeycomb lattice at all fillings and temperatures. The signal is somewhat diminished from that of Kr as can be seen in the top part of Fig. 3, simply due to the lower atomic number of Ar. Also the background from inevitable flakes of graphite in the (002) orientation at 1.89 $Å^{-1}$ is considerably higher, but the incommensurate Ar peak can nevertheless clearly be measured with high resolution. The full line is the calculated profile corre-

sponding to L = 750 Å. We plan to study the phase diagram and in particular the melting of this incommensurate phase in the near future.

B. Phase Transitions of the Smectic A Phase of the Liquid Crystal 4.08

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Liquid crystals consist of long rod-like molecules, typically containing two benzene-rings in the centre and aliphatic hydrocarbon chains in each end. In the present case one hydrocarbon chain has 4 members, the other 8, and the linkage between the former tail and the benzene rings is via an oxygen atom, hence the name 4.08.

In the smectic A phase [8] the molecules are arranged in layers with the molecular axis normal to the layers. There is a well defined layer repetition distance, but liquid-like order within each layer. The order parameter for this phase is a one-dimensional density wave characterized by an amplitude and a phase. In some materials the order parameter vanishes practically continuously as the temperature is raised towards the transition temperature T_c to the nematic phase where the layer ordering is destroyed, but an overall orientation of the molecules is maintained. Smectic-A-like fluctuations occur of course in the nematic matrix within a correlation range ξ (or rather $\xi_{\parallel}$ along the axis and $\xi_{\perp}$ perpendicular to the axis) and ξ diverges as $T \to T_c$. A phenomenological Landau analysis shows readily that the transition is isomorphous to that of superconductivity in metals [9].

In lowering the temperature through the smectic A phase 4.08 undergoes a phase transition to the B-phase, which has been interpreted to be another smectic phase with short range order within the planes. Recently, however, two of us (D.E.M [10] and

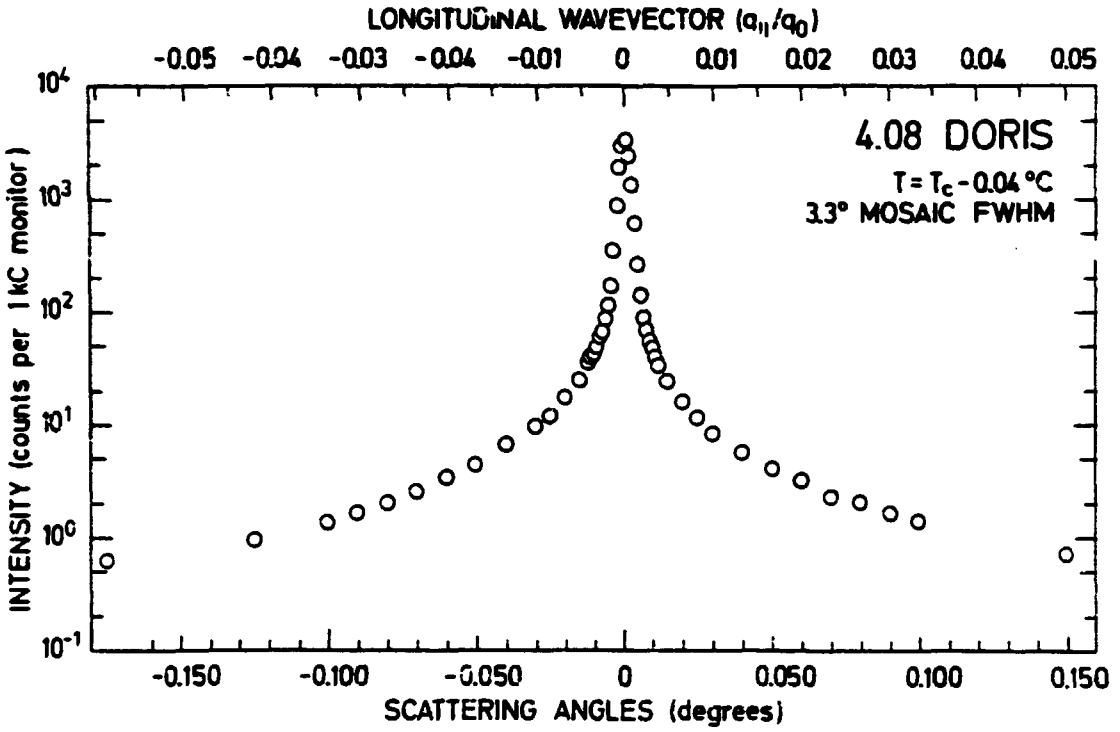

Fig. 4. Line shape of the (001) "Bragg" reflection in the smectic A phase. The data, extending over 4 orders of magnitude in intensity are consistent with a cross section diverging as $q_{\parallel}^{-2+\eta}$ rather than a delta function at $q_{\parallel} = 0$ superimposed on some thermal diffuse background. This divergence corresponds to algebraic decay of the layer stacking order, a phenemenon to be expected also in 2-dimensional crystals due to thermal fluctuations.

P.S.P. [11] and coworkers) showed independently by high resolution X-ray diffraction techniques that the B-phase was nothing but a genuine 3-dimensional crystalline phase.

The smectic A phase itself does not have true long range order. This peculiarity comes about for very much the same reason that thermal fluctuations inhibit true positional long range order in a 2-dimensional crystal [12].

Consequently the Bragg scattering from the stack of layers, the (001)-reflection, is not a delta-function [13] but is rather to be described by a divergence $q^{-2+\eta}$, where q is the difference between the wavevector transfer and the (001) point, and η is a small parameter depending on two elastic constants describing longitudinal and transverse fluctuations of the layer arrangement.

This phenomenon of quasi-long range order was observed by one of us (J.A.-N.) and his collaborators recently [14] in a so-called bi-layer smectic A material. Here the molecule does not have aliphatic chains in both ends but rather a polar head and a tail, and molecules pair by sticking their polar heads together. There is no fundamental reason for the quasi-long range order to be associated with bi-layer type materials, so we felt it was of some importance to observe it also in single layer materials. Furthermore in 4.08 the $q^{-2+\eta}$ divergence should change in the B phase to a true Bragg peak plus thermal diffuse scattering.

After this exposition of our motivations for the experiment let us give an example of experimental results on the quasi-long range order obtained with synchrotron radiation. A sharp, narrow resolution was obtained by using channel-cut crystals as monochromator and analyzer. The profile of the (001) reflection is shown in Fig. 4 showing the logarithm of the intensity versus wavevector deviation q.

With the synchrotron source the quasi-long range order peak can be traced over 4 decades in intensity and much further out in reciprocal space than in the original experiment [14] at a 10 kW rotating anode. We have also measured the profile in the B phase as well as the critical fluctuations in the nematic to smectic A transition. The results will be published shortly.

C. Critical Fluctuations in the Nematic-Smectic A-Reentrant Nematic Phase Transitions in Liquid Crystal Mixtures

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In the previous section we discussed the phase transition from the high temperature nematic phase to the low temperature smectic A phase. In lowering the temperature further one may obtain the nematic phase again, the re-entrant nematic phase. This striking phenomenon occurs in some bi-layer compounds at elevated pressures [15], in mixtures of different molecules [16] and even in pure substances at ambient pressure [17].

In a recent study [18] of the viscosity in the ternary mixture of heptylcyanobiphenyl (7CB), octyloxycanobiphenyl (80CB) and pentylcyanoterphenyl (5CT) in the ratio of 73:18:9 by weight it was noted that the re-entrant phase was not a metastable phase as found in many other mixtures and the phase diagram versus relative concentration of 80CB and 7CB [19] shown in the lower part of Fig. 6 indicates an interesting point at 76.8% 7CB where the material barely becomes smectic A at any temperature.

Our aim was to study the critical behaviour of the fundamental quantity driving the phase transition, the correlation range ξ, in both the nematic and the re-entrant nematic phase and for a number of concentrations encompassing the critical concentration of 76.8% 7CB. In order to measure the critical divergence of ξ up to several hundred times the molecular length high resolution X-ray diffraction must be used, and we have established the technique for such measurements a couple of years ago using perfect crystal techniques and a rotating anode as the X-ray source [20].

In the present case the virtue of synchrotron radiation is primarily the fast data accumulating rate. Within one week we were able to obtain high quality data for 6 mixtures at both phase transitions corresponding to 12 experiments of the kind described in Ref. 20.

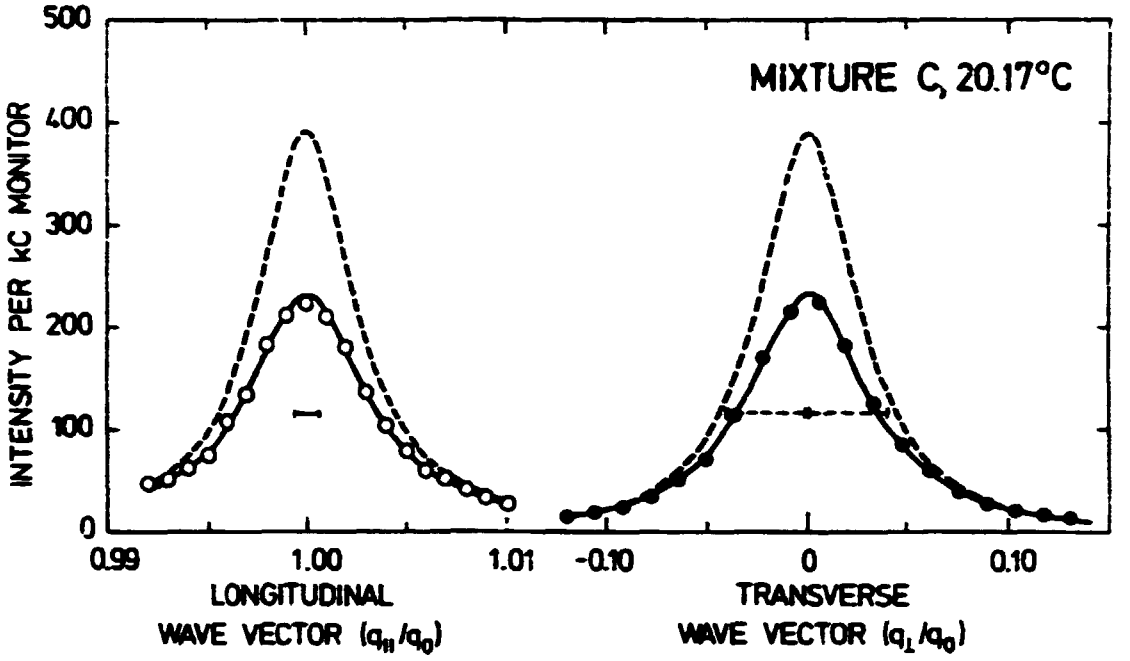

Fig. 5. Longitudinal and transverse scans through the (0,0,1) reciprocal lattice point show the smectic A fluctuations in the nematic phase. Full lines are least squares fit of the cross section (dashed curve) folded with the resolution.

A summary of our results is given in Fig. 6. Let us first discuss one temperature point on the curve labelled C corresponding to a certain concentration. A longitudinal scan and a transverse scan through the (001) point of reciprocal space are shown in Fig. 5. Both peaks become more intense and narrow as the temperature is lowered towards the transition temperature T_c. The width of the transverse peak exhibits a distinct kink at $T = T_c$, the limiting width being the mosaic width of the smectic A phase. The kink determines the critical temperature within a few millidegrees. The full line profile of the longitudinal scan is a best fit of a Lorentzian cross section (dashed line) of full width $2/\xi$ at half maximum folded with the experimental resolution. All of the data in Fig. 5 are thus recorded to a single point, $\xi^{-1}(c)$, plotted on curve C in Fig. 6.

The data at a concentration slightly larger than the limiting concentration of 76.8% for obtaining the smectic A phase (curve B) are particularly amusing. Smectic A fluctuations clearly occur in the high temperature nematic phase with a correlation range growing with decreasing temperature. But the growth never makes it to a divergence and when the temperature is lowered below $T=15.5^{o}C$ the correlation range starts to <u>decrease</u> with decreasing temperatures.

In summary, the critical behaviour of ξ vs. $|T-T_c|$ depends on the path in the phase diagram: The more tangential the path is to the phase boundary the more gentle is the temperature dependence of ξ. A phenomenological Landau theory for this behaviour has already been outlined by J. Prost [21], and we plan to analyze our data along these lines shortly.

4. FUTURE INSTRUMENT IMPROVEMENTS

Basically the triple-axis spectrometer set-up has been developed to the point where it can readily be used for experiments. In particular it is worth noting that although only a horizontal aperture of 0.1-0.2 mrad is used the intensity is sufficiently

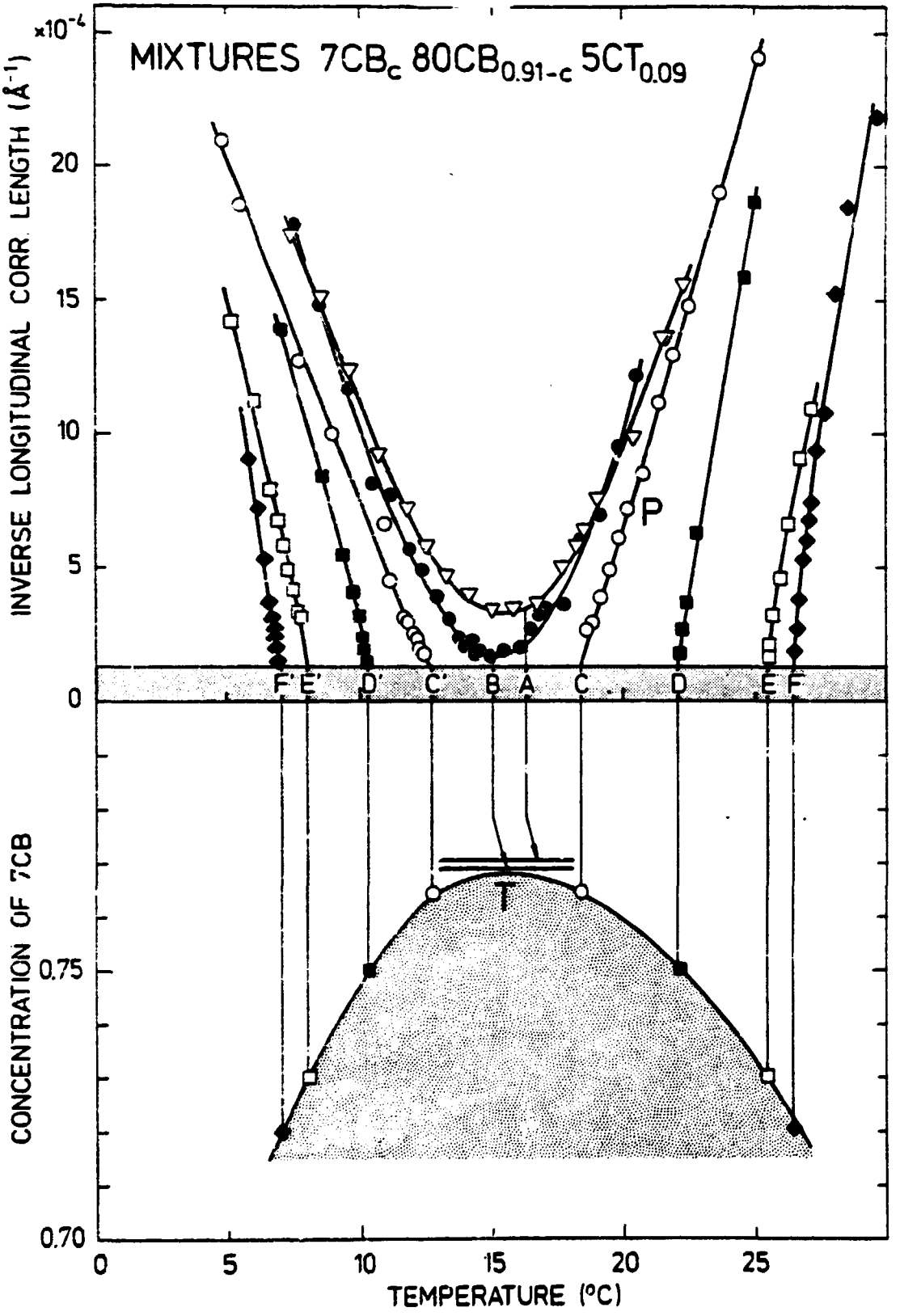

Fig. 6. Top: Correlation range of smectic A fluctuations in the nematic phase in mixtures versus temperature. The grey area indicates the resolution limit of the correlation range.
Bottom: Phase diagram of mixtures. The grey area denotes the smectic A phase.

above conventional sources to enable exploration of new areas of research.

It is nevertheless tempting to think about utilizing a larger part of the available 1 mrad horizontal aperture. One possibility is focusing by means of a curved mirror, but that is probably not feasible in the present beam line. Another possibility is to use a double monochromator in the vertical plane and let the first crystal be a Si wafer bent around the horizontal axis. We do not see any serious difficulty in building such a monochromator, but in order to utilize it in a diffraction experiment the spectrometer must be modified to operate in the vertical plane and here the proximity of the neighbouring beam lines and the wall of the hutch may present a serious obstacle, cf. Fig. 1.

The immediate future involvement in the instrumentation is (i) installation of the permanent beam line D4 in HASYLAB, (ii) motorization of the floor level spindels L1, L2 and L3 together with a development of an ionization chamber arrangement in the white beam, so the instrument can be adjusted to beam height within a few minutes and the beam position constantly monitored, (iii) a rugged horizontal translation of the monochromator axis across the white beam, (iv) remote control of the slit system after the monochromator drum.

5. CONCLUSIONS

Our experience from a 3-week period with the storage ring operating 12 hours a day in a mode dedicated to synchrotron radiation is very encouraging. Once the dedicated mode had been established the beam was very stable within each run, one or two extra fillings of the storage ring during a 12-hour period were sufficient and the beam position was quite reproducible from one shift to the next.

It is important to establish a fast line-up procedure with as many of the adjustments as possible under remote control. A T.V. camera for watching movement of the stepping motors as well as for seeing the beam on a fluorescent screen turned out to be quite convenient.

Generally one must advise that the experiments be well prepared and possibly carried out crudely at a rotating anode beforehand because the 12-hour beam time, typically requiring a 15-hour on-site time, with a phenomenal data acquisition rate leaves very little time for wise planning of the experimental strategy while the experiment is being executed.

6. ACKNOWLEDGMENTS

We are grateful to HASYLAB for permission to use the facility. The smooth collaboration with the staff at HASYLAB is gratefully acknowledged. In particular we appreciate the efforts of Dr. G. Materlik for getting us a prompt start on synchrotron X-ray diffraction and for helping us in solving enumerable small and bigger practical problems as we proceeded.

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